Tensile bond strength of a lithium-disilicate pressed glass ceramic to dentin of different surface treatments

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The effects of desensitizer, disinfectant, saliva, blood, and hydrogen peroxide on the tensile bond strength between adhesive and ceramic as well as between adhesive and dentin were examined. Sixty 7×3 mm pressed ceramic discs of IPS e.max were fabricated and randomly assigned to six groups of different dentin surface treatments (control, desensitizer, disinfectant, saliva, blood, and hydrogen peroxide). Representative samples of fractured specimens were observed by SEM (scanning electron microscopy). There were significant differences between the control group and saliva, blood, and hydrogen peroxide groups ($p<0.05$). However, there were no significant differences between any other dentin surface treatment groups ($p>0.05$). Results of this study suggested that only saliva, blood, and hydrogen peroxide influenced the tensile bond strength between dentin and ceramic.

Keywords: Dental ceramics, Adhesive cements, Tensile strength

INTRODUCTION

It has becoming increasing popular for all-ceramic dental restorations to combine an esthetic but weak porcelain veneer with a strong ceramic core. Veneering porcelains typically consist of a glass and a crystalline phase of fluoroapatite, aluminum oxide, or leucite. Veneering a lithium disilicate, aluminum oxide, or zirconium oxide core with glass allows dental technicians to customize restorations in terms of form and esthetics. The success of these systems hinges on preventing failures between the restorative material and the dental substrate^{1–5}.

Notably, good adhesion between the tooth and restorative material is recognized to play a pivotal role in the success of dental restorations^{1,6}. When bonding ceramic to tooth substrates, it is important to ensure optimal bond strength at two different interfaces — namely, the dentin-adhesive and ceramic-adhesive interfaces, because these two interfaces determine the overall, final bond strength of the restoration⁷. Moreover, durable bonding between dentin and adhesives is important for avoiding detachment of restorations and for preventing microleakage, secondary dental caries, and tooth fractures¹⁰. At the ceramic-adhesive interface, *in vitro* studies have shown that reliable bonding could be achieved by acid-etching the ceramic surface and applying a silane coupling agent^{8,9}. At dentin-adhesive interfaces, it has been shown that reliable bonding could be achieved by dentin surface treatments. However, the dentin-adhesive bond is not as strong as that between ceramic and dentin because dentin is biologically active and complex^{11,12}.

On factors that may affect the chemical interaction between restorative materials and dentin, and perhaps even the sealing and bonding properties of luting agents, they include the ingredients of desensitizers,

antibacterial solutions, and hydrogen peroxide. Besides, saliva and blood contamination may also create a thin film on the dentin surface that hinders adhesive penetration into the dentinal tubules^{10,13,14}. In the wake of these concerns, the present study evaluated the effects of various dentin surface treatments (desensitizer, disinfectant, saliva, blood, hydrogen peroxide, no treatment [wet dentin]) on the tensile bond strengths of the adhesive-dentin interface and adhesive-ceramic (lithium disilicate glass ceramic) interface. The null hypothesis was that the tensile bond strengths of both interfaces would be similar for all dentin surface conditions.

MATERIALS AND METHODS

The materials used in this study are presented in Table 1.

Ceramic disc preparation

Sixty 7×3 mm pressed lithium disilicate glass ceramic discs of IPS e.max (Ivoclar Vivadent AG, Schaan, Liechtenstein) were fabricated using the lost wax technique, and ingots were injected into an EP 600 furnace (Ivoclar Vivadent AG, Schaan, Liechtenstein).

Disc surfaces were ground with 220-, 360-, and 600-grit silicon carbide sandpapers to standardize the bonding surfaces. Ground ceramic discs were air-abraded with 50- μ m Al₂O₃ particles (Korox, Bego, Bremen, Germany) for 14 seconds from a distance of approximately 10 mm at 400 kPa with a sandblasting device (Ar-Ge Dental, Denizli, Turkey). The discs were then cleaned in distilled water for 10 minutes in an ultrasonic bath (Healthsonics, Livermore, California, USA) to ensure contaminant-free surfaces. The bonding surfaces of the ceramic discs were etched with 37% phosphoric acid (K-Etchant Gel, Kuraray, Tokyo, Japan) for 5 seconds, rinsed for 30 seconds with a

Table 1 Materials used in the study

Name	Manufacturer	Ingredients	Lot number
IPS e.max	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate glass ceramic	H36067
Clearfil Esthetic Cement	Kuraray, Tokyo, Japan	Paste A: Bis-GMA, TEGDMA, methacrylate monomers, silanated glass filler. Paste B: Bis-GMA, TEGDMA, methacrylate monomers, silanated glass filler, colloidal silica, benzoyl peroxide, dl-camphorquinone.	08000101126
Clinpro White Varnish desensitizer	3M ESPE, St. Paul, MN, USA	Sodium fluoride ions, modified resins	M12190
Farhex	Santa Farma, Istanbul, Turkey	Chlorhexidine gluconate, benzydamine hydrochloride, aspartame, ethyl alcohol	1808

water spray, air-dried, and then silanated with a silane coupling agent (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) for 60 seconds.

Tooth preparation

Sixty caries-free unrestored human third molars, which had been stored in 0.5% Cloramin-T solution at 4°C for up to 1 month after extraction, were selected as tooth specimens. Soft tissues were removed with a scaler (H6/H7 Scaler, Hu-Friedy, Chicago, USA). After cleaning the teeth with pumice, each tooth was embedded in an autopolymerizing acrylic resin (Palapress Vario, Heraeus Kulzer GmbH, Wehrheim, Germany) in a cylindrical plastic mold of 20 mm height and 20 mm diameter. Enamel was removed by cutting off the crown horizontally at the middle, exactly at the top of the pulp chamber, using a low-speed diamond saw (Minitom, Struers, Copenhagen, Denmark). Deep dentin was chosen in this study to favor the diffusion of adhesive into the demineralized dentin. This is because the number of tubules in coronal dentin increases significantly from the middle to the deep layer—nearest to the pulp, thus providing the best conditions for resin monomer infiltration. A standard smear layer was produced by wet-sanding the dentin surface with 600-grit silicon carbide sandpaper for 40 seconds.

Experimental design

The 60 prepared specimens were randomly assigned to six groups of different dentin surface treatments ($n=10$ each):

Group 1 (control): Wet dentin. No contaminant was added.

Group 2: Clinpro White Varnish desensitizer (3M ESPE, St. Paul, MN, USA) was applied according to manufacturer's instructions. Dentin surface was rinsed with a water spray for 10 seconds and blotted with absorbent paper.

Group 3: Farhex (2%, 1 mL) disinfectant solution was applied for 20 seconds. After rinsing with a

water spray for 10 seconds, excess water was removed with absorbent paper.

Group 4: Saliva was applied with a microbrush for 20 seconds, rinsed for 10 seconds, and blotted with absorbent paper.

Group 5: Fresh capillary human blood (supply from a single donor) was applied with a micropipette for 20 seconds, rinsed for 10 seconds, and blotted with absorbent paper.

Group 6: Hydrogen peroxide (H_2O_2) applied with a micropipette for 20 seconds, rinsed for 10 seconds, and blotted with absorbent paper.

Bonding procedures

A dual-polymerizing resin cement was used (Clearfil Esthetic Cement, Kuraray, Tokyo, Japan) as the adhesive. Equal amounts of ED Primer II Liquids A and B were mixed and applied to the dentin surface for 30 seconds, and then the surface was thoroughly air-dried. Clearfil Esthetic Cement pastes A and B were mixed and then applied to the ceramic surfaces using a dispenser syringe. A special loading device was designed to apply a constant load of 9.2 N to the ceramic discs. This load was used to create a uniform resin luting layer of approximately 100 μ m, so as to simulate the film thickness employed for all-ceramic crowns. Initial light curing was performed for 10 seconds. Excess cement was removed using a dental probe. The luting agent was polymerized from each direction (mesial, distal, buccal, lingual, occlusal) with an LED curing device for 40 seconds (light intensity: $-1,000 \text{ mW/cm}^2$; Elipar FreeLight 2 LED Curing Light, 3M ESPE, MN, USA).

Tensile bond strength test

Following storage in distilled water at 37°C for 24 hours, the specimens were thermocycled for 6,000 cycles between $5\pm 2^\circ\text{C}$ and $55\pm 2^\circ\text{C}$, with a dwell time of 20 seconds and a transfer time of 5 seconds. The thermocycling process was completed in 4.5 days. If a spontaneous interfacial debonding occurred during the

thermocycling process, the corresponding bond strength was recorded as 0 MPa.

After the thermocycling process, each specimen was mounted on the lower fixture of a universal testing machine (Model 6022, Instron Corp, Norwood, USA) and an orthodontic wire connected to the upper fixture. Bonded dentin ceramic specimens were then subjected to a tensile force at a crosshead speed of 2 mm/min until decementation occurred. Tensile bond strengths were calculated using the following formula:

$$\text{Tensile bond strength (MPa)} = P/A$$

where P is the maximum force (N) and A is the interfacial area (mm²).

Stereomicroscope and scanning electron microscope (SEM) examination

After tensile bond strength testing, the dentin surfaces of the debonded specimens were examined with a stereomicroscope (Wild M3B, Heerbrugg, Switzerland) at 25× magnification to identify bond failure modes. Failure modes were classified as follows:

- Adhesive failure at dentin-resin interface (A/D-R): Dentinal tubules were observed to be open and no broken resin was apparent on the intertubular dentin surface;
- Adhesive failure at resin-ceramic interface (A/R-C); and
- Cohesive failure within resin (C/R).

For SEM analysis, two specimens were randomly selected from each group for this purpose. The debonded specimens from each group were sputter coated (Bal-Tec SCD 050 Sputter Coater, Bal-Tec AG, Balzers, Liechtenstein) with gold and observed with a scanning electron microscope (LEO 440, Leica-Zeiss, Cambridge, UK).

Statistical analyses

All data sets were subjected to normality tests using the Kolmogorov–Smirnov method, and data were presented as medians and 25th and 75th percentiles (for skewed data). The nonparametric Kruskal–Wallis test was used to compare the groups, and Dunn's multiple comparison test was used to evaluate the subgroups. *p* values of <0.05 were deemed statistically significant. All analyses were performed using a statistical package for scientists, SigmaStat (Aspire Software International, Washington, USA), Windows version 3.10.

RESULTS

Specimens which debonded during thermocycling were added to statistical analysis as 0 MPa. Medians and 25/75 percentiles of the measured tensile bond strengths are shown in Table 2. Kruskal–Wallis test revealed significant differences between the groups (*p*<0.05), and Table 3 presents the results of Dunn's multiple comparison test. There were significant differences between Groups 1 and 4 (*p*<0.05), between Groups 1 and 5 (*p*<0.05), and between Groups 1 and 6

Table 2 Medians and 25/75 percentiles of the tensile bond strength (MPa) of dentin surface treatment groups

Groups	<i>n</i>	Median	25%	75%
Group 1	10	22.61	19.42	28.74
Group 2	10	16.87	10.30	28.25
Group 3	10	8.82	6.28	14.72
Group 4	10	2.35	0	7.46
Group 5	10	1.71	0	19.52
Group 6	10	4.36	0	10.01

Table 3 Comparison (*p* values) between dentin surface treatment groups

Dunn's multiple comparison test	<i>p</i> values
Control/desensitizer	>0.05
Control/chlorhexidin gluconate	>0.05
Control/saliva	<0.05
Control/blood	<0.05
Control/H ₂ O ₂	<0.05

Table 4 Failure mode data

Groups	<i>n</i>	A/D-R	C/R	A/R-C
Group 1	10	3	6	1
Group 2	10	5	4	1
Group 3	10	7	3	–
Group 4	10	8	2	–
Group 5	10	9	1	–
Group 6	10	10	–	–

A/D-R: Adhesive failure at resin-dentin interface; C/R: Cohesive failure of resin; A/R-C: Adhesive failure at resin-ceramic interface.

(*p*<0.05). There were no significant differences between any other groups.

Failure modes of the six surface treatment groups as identified using a stereomicroscope and the number of occurrences thereof are shown in Table 4. In the control group, there were three occurrences of adhesive failure at the adhesive-dentin interface, but it varied between five and 10 occurrences in the other surface treatment groups.

The SEM images of representative fractured

Fig. 1 No contaminant was applied in the control group. SEM image of a smear layer covering the dentin surface (A/R-C). Magnification: 500×.

Fig. 3 SEM image of dentin surface treated with chlorhexidine gluconate. Smear layer covered the dentin surface (A/R-C). Magnification: 500×.

Fig. 2 SEM image of dentin surface treated with desensitizer. Smear layer was partially removed, and both residual resin and tubular orifices were observed (C/R). Magnification: 500×.

Fig. 4 SEM image of dentin surface contaminated with saliva. Smear layer was removed, and the tubular orifices were observed to be open all over the bonding area (A/D-R). Magnification: 500×.

specimens from the six dentin surface treatment groups are shown in Figs. 1–6. In the control group and Group 3 (Figs. 1 and 3 respectively), resin covered the dentin surface and their failure modes were determined as A/R-C adhesive failure. In Group 2 (Fig. 2), both residual resin and tubular orifices were observed, and the failure mode of specimen was determined as C/R cohesive failure. In Groups 4, 5, and 6 (Figs. 4, 5, and 6 respectively), the tubular orifices were observed to be open over the bonding area and the failure modes of specimens were determined as A/D-R adhesive failure.

DISCUSSION

A durable and predictable bond strength between dental materials and tooth substrates is critical to clinical success¹⁵⁾. In this respect, it is very important to detect and remove contaminants from the dentin surface to ensure the long-term success of dental restorations¹⁶⁾. Thus, in this study, the effects of desensitizer, disinfectant, saliva, blood, and hydrogen peroxide contamination on adhesive-dentin bonding were investigated by simulating a clinical try-in procedure. The results obtained in this study did not support the hypothesis that the tensile bond strengths of both adhesive-dentin and adhesive-ceramic interfaces

Fig. 5 SEM image of dentin surface contaminated with human blood. Smear layer was removed, and the tubular orifices were observed to be open all over the bonding area (A/D-R). Magnification: 500×.

Fig. 6 SEM image of dentin surface treated with H₂O₂. Smear layer was removed, and the tubular orifices were observed to be open all over the bonding area (A/D-R). Magnification: 500×.

are similar for all dentin surface conditions.

Water storage, combined with thermocycling at regular intervals, was used to simulate aging in this study¹⁷. Thermocycling resulted in a rapid increase in the rate of water absorption into the composite at higher temperatures, causing hydrolysis of the silane bond. When coupled with a contaminated dentin surface, thermocycling decreased the bond strength by weakening the resin structure^{18,19}. Owing to this weakening effect, dentin surfaces which were treated with different contaminants debonded during thermocycling, except the control group to which no contaminant was added.

Desensitizing agents are used on exposed dentin surfaces to prevent dentin hypersensitivity²⁰. In this study, Clinpro White Varnish, which is a new fluoride-containing desensitizer, was used to treat the dentin surface (with acid-etching not before but after application, as per manufacturer's recommendations). The formation of a hybrid layer could be seen in SEM images (Figs. 1 and 2). The acid-etching procedure removed the smear layer and depleted fluoride ions from the dentin surface, allowing the formation of a hybrid layer. Resin monomers penetrated the tubules, forming resin tags with a jagged-like feature. On the other hand, the deposition of fluoride ions on the dentin surface could neutralize the etching procedure and inhibit the formation of a hybrid layer. These effects of fluoride deposition on the dentin surface might subsequently interfere with the bonding procedure, which meant that fluoride-containing desensitizers might decrease dentin bond strength^{20,21}. However, our results with Clinpro White Varnish desensitizer in this study were not in agreement with those of previous studies^{20,21}, which meant that more research is needed in this area.

In Group 3, the dentin surfaces of specimens were

treated with chlorhexidine gluconate disinfectant. After filling a cavity, bacteria that remain behind may cause secondary caries under the restoration and further damage the tooth²². For this reason, chlorhexidine gluconate is widely used as an antimicrobial agent for disinfection before the placement of restorations. Chlorhexidine gluconate, which binds to the amino acids in the dentin and which is able to kill bacteria for several hours, makes it an effective antimicrobial agent²³. In this study, our results were consistent with those of previous studies which showed that chlorhexidine gluconate application prior to acid-etching had no adverse effect on both adhesive-ceramic and adhesive-dentin interfaces^{24,25}. Moreover, SEM observation seemed to suggest that chlorhexidine gluconate application might also lead to better preservation of naked collagen fibrils as they were sealed by a layer of adhesive resin (Fig. 3)¹³.

However, the benefit(s) of using chlorhexidine gluconate should be further weighed and examined against the side effects which limit its widespread use, such as brown staining of the teeth, an increase in calculus deposits, and difficulty in completely masking its taste when used as a rinse. Despite these concerns and drawbacks, using chlorhexidine gluconate to complement restorative procedures does not typically lead to these negative effects²⁵. To justify the safe use of chlorhexidine gluconate for dental restorative treatments, further studies are needed to analyze the beneficial and harmful effects of using chlorhexidine gluconate solutions with dental adhesive systems, and the influence of chlorhexidine gluconate application on bonding stability over time.

Saliva is 99.4% water, and the remaining composition comprises macromolecules (such as proteins, glycoprotein sugars, and amylase), inorganic particles (such as urea, amino acids, fatty acids, and

free glucose), bacteria, and food debris. After saliva contamination, non-covalent adsorption of salivary protein occurs on the surfaces of both the tooth and restorative material, creating an organic coating that cannot be removed by rinsing with tap water¹⁷. In the present study, salivary contamination (Group 4) was found to significantly decrease the tensile bond strength between dentin and ceramic, hence agreeing with the results of other reports^{14,17,26}. Webster *et al.*²⁷ found that when saliva contamination occurred after applying a primer to dentin, reapplication of the primer after drying the saliva provided a good bond to the underlying primer layer. In addition, Eiriksson *et al.*²⁶ reported that drying the saliva quickly resulted in a significantly stronger bond than drying the saliva slowly, which was probably due to reduced saliva film thickness. In the present study, SEM observation of saliva contamination revealed a smooth surface on the specimens, which seemed to suggest that reduced bond strength was probably due to a lack of interaction of the resin increment with the contaminated dentin surface (Fig. 4).

Prior to the placement of restorations in a clinical setting, there is a relatively high risk of blood contamination on the adherent dentin surface, especially for crown margins in the gingival area²⁸. In the present study, the blood-contaminated specimens were rinsed, but there remained a significant difference between the blood contamination group (Group 5) and the control group. Blood dried on the dentin surface caused a catastrophic decrease in bond strength at ceramic-resin interface, which was in agreement with published results^{28,29,30}. SEM observation (Fig. 5) revealed a smooth surface on the specimens when compared to the other surface treatments (Figs. 1, 2, 3, and 6), and which probably resulted in reduced bond strength due to the lack of interaction of the adhesive resin with the contaminated surface.

Adhesive systems are sensitive to saliva and blood contamination. This is attributable to the depositing of macromolecules of these contaminants into the dentinal tubules. In particular, blood contamination could reduce adhesive-dentin bond strength to a greater degree than salivary contamination, as its high protein content and macromolecules such as fibrinogen and platelets create a thin film on the dentin surface which hinders adhesive penetration into dentinal tubules³¹.

Hydrogen peroxide is one of the most common disinfectants and coagulant materials used in dentistry³². In this study, the tensile bond strength value of Group 6 (hydrogen peroxide) was statistically lower than the control group, which was in agreement with that of a previous study³³. It was probable that hydrogen peroxide which remained on the dentin surface altered surface morphology, causing a reduction in dentin-adhesive bond strength. Moreover, hydrogen peroxide decomposes into water and oxygen, with the latter penetrating rapidly through the porosities of dentin organic matrix, thereby exerting its detrimental effects³².

To compare the results of studies which have investigated contamination in adhesive restorations is a complex undertaking. Differences in the types of adhesive system, contamination period, substrate, and blood type reflect and underline a lack of methodological standardization. In addition, blood, salivary, and gingival fluid contamination typically occur concomitantly³¹.

CONCLUSION

Within the limitations of this *in vitro* study, this paper concluded that saliva contamination, blood contamination, and hydrogen peroxide application influenced the tensile bond strengths of adhesive-ceramic and adhesive-dentin interfaces, whereas no such detrimental effect was observed with Clinpro White Varnish and chlorhexidine gluconate applications.

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